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STOPPING POWER EFFECTS ON RUTHERFORD BACKSCATTERING
ANALYSIS IN THICK TARGETS(U) ARMY BALLISTIC RESEARCH
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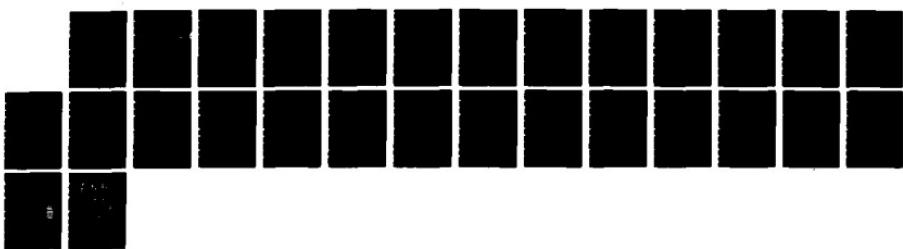
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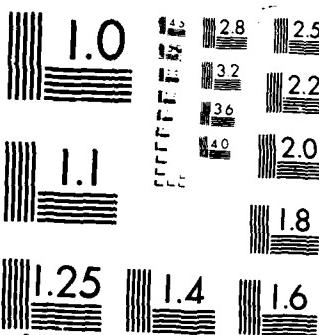
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STOPPING POWER EFFECTS ON
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ANALYSIS IN THICK TARGETS (U)

DR. ANDRUS NIILER

JULY 1987

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US ARMY BALLISTIC RESEARCH LABORATORY
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1. INTRODUCTION

Ion Beam Analysis (IBA) has become a very useful technique for the purpose of determining detailed information about materials and their surfaces. A large body of literature has accumulated describing this method and detailing many of its applications and advantages. An excellent review can be found in Ref. 1. Some significant applications of IBA relevant to Army research are the analysis of thin protective coatings, coating/substrate interfaces, erosion surfaces in conventional and EM gun barrels, corrosion studies, surface modification by ion implantation and evaluation of powder surface impurities. Some of these applications are discussed in Refs. 2-5. A short description of the IBA methods known as Rutherford Backscattering (RBS) and Nuclear Reaction Spectroscopy (NRS) follows.

RBS and NRS are surface analysis techniques which are capable of determining both the elemental concentrations and their depth profiles in the outer several microns of a solid surface. The method requires placement of the analysis sample into a vacuum chamber where it is bombarded by a beam of energetic ions such as alpha particles or deuterons. As the incident ions interact with target nuclei, they undergo either elastic, billiard ball like, scattering or nuclear reactions. In this paper the former will be referred to as Rutherford Backscattering Spectroscopy (RBS) and the latter as Nuclear Reaction Spectroscopy (NRS). When the elastically scattered beam particles or reaction products leave the sample surface, their energies are measured by a solid state detector placed at a backward (i.e. 160°) angle to the beam and the energy spectra are recorded.

In RBS, the resultant spectra are slowly varying distributions of scattered particle yields as a function of particle energy. The shapes of these RBS distributions are determined almost wholly by the stopping power of the sample medium to the ion and the ion-sample interaction probability. The latter is generally given by the Rutherford cross section (hence the name Rutherford Backscattering) and the former is generally a semi-empirically determined function based mostly on the evaluation of large amounts of stopping power data. RBS distinguishes between sample elements by the amount of

¹Chu, W. K., Mayer, J. W., and Nicolet, M. A., Backscattering Spectrometry, Academic Press, New York, 1978.

²Niiler, A., "Ion Beam Methods Applied to Interior Ballistic Studies," IEEE Trans. Nucl. Sci., Vol. NS-28, pp. 1834, 1981.

³Niiler, A., Birkmire, R., and Caldwell, S. E., The Effects of Propellant Burn on the Surface Composition of Gun Steel, ARBRL-TR-02380, Ballistic Research Laboratory, Aberdeen Proving Ground, MD, 1981.

⁴Jamison, K. A., Burden, H. S., Marquez-Reines, M., and Niiler, A., Analysis of Rail Gun Bore Residue, ARBRL-TR-02554, Ballistic Research Laboratory, Aberdeen Proving Ground, MD, 1984.

⁵Proceedings of the Ion Implantation for Army Needs Workshop, AMMRC MS 84-1, Army Materials and Mechanics Research Center, Watertown, MA, 1984.

energy imparted to the backward moving particle which is related to the mass of the sample nucleus from which it scattered. This kinematic dependence of scattered energy on incident and target masses, m and M , the scattering angle θ , and the incident and scattered particle energies E_0 and E , is shown in Ref. 6 and is given by

$$E = E_0 \left(\frac{m \cos \theta + \sqrt{M^2 + m^2 \sin^2 \theta}}{m + M} \right)^2.$$

The consequence of this equation is that target identification improves when either the incident mass increases or the target mass decreases, i.e. the ratio, m/M , increases.

In NRS, the interaction between incident and target particles is an exothermic nuclear reaction by which some of the system mass is converted to energy which is in turn imparted to the outgoing particle. This outgoing particle has a different identity than the incident one. In the case of the BRL experiments, the incident particles were deuterons and the outgoing particles were either protons or alphas. Deuterons were chosen because they have reactions with the light nuclei such as carbon, nitrogen and oxygen, allowing these elements to be both identified and profiled.

One of the most significant features of RBS and NRS methods in material surface analysis is the capability to make, non-destructively, absolute measurements of elemental concentrations without the need for calibration samples. Even though calibration samples are not required, basic data such as atomic stopping powers, $S(E)$, scattering/reaction cross sections as well as experimental parameters like beam charge and a well defined geometry are still necessary. By far the most important requirements in the measurement of absolute elemental concentrations or coating thicknesses are the elemental stopping powers and scattering or reaction cross sections. All calculations which are used to extract concentration or thickness information from RBS/NRS raw data, from the simplest and most easy to use⁶ to the most complex,^{7,8} depend very strongly on these two factors.

At the energies where most RBS work is performed, alpha particles at 2-3 MeV or deuterons at about 1 MeV, the scattering cross section is always assumed to be Rutherford. This assumption is generally good to a few percent

⁶Foti, G., Mayer, J. W., and Rimini, E., Ion Beam Handbook for Material Analysis, Academic Press, New York, 1977.

⁷Doolittle, L. R., "Algorithms for the Rapid Simulation of Rutherford Back-scattering Spectra," Nucl. Inst. Meth., Vol. B9, pp. 344, 1985.

⁸Niiler, A., Birkmire, R., and Gerrits, J., PROFILE: A General Code for Fitting Ion Beam Analysis Spectra, ARBRL-TR-02233, Ballistic Research Laboratory, Aberdeen Proving Ground, MD, 1980.

except for deuterons on the light elements such as carbon, nitrogen and oxygen where large deviations from Rutherford occur to energies as low as 0.5 MeV. The uncertainties on the non-Rutherford elastic cross sections or the deuteron induced nuclear reaction cross sections on the light elements are in the 10% range. Typically, the cross section uncertainties lead to equivalent uncertainties in the concentrations of the elements deduced from the profiling analysis. However, if cross section uncertainties are combined with uncertainties in the stopping powers, more serious effects can occur. In these cases, it is possible for the RBS analysis to require unphysical amounts of elements whose detection is either very difficult or impossible.

If the analysis experiment is constrained to regions where the cross sections are all Rutherford, then the ability to deduce absolute values of layer thicknesses and elemental concentrations depends completely on the accuracy of stopping powers. Much work has gone into the measurement and evaluation of elemental stopping powers at energies used in IBA experiments. However, serious discrepancies continue to crop up between the generally used values^{9,10} and newly measured data,¹¹ for instance. In this particular case, the discrepancies are 15% or more for hydrogen ions on common metals like nickel and copper, in the region of the stopping power maximum. Although this region at around 100 KeV/amu may not enter into RBS calculations when 2 to 3 MeV alphas are used and only thin surface layers are analyzed, it does become very important when RBS and NRS is done with 1 MeV deuterons and the depth of the probed layers exceed 0.5 mg/cm².

In this work, the effect of Al, Ni and Cu stopping power uncertainties in the region of the maximum of the stopping power curve on 1 MeV deuteron RBS analysis will be examined. The use of thick target RBS data along with careful simulation of the experiment as a method for easy and accurate stopping power determination will also be discussed.

2. EXPERIMENTAL PROCEDURES

A 1.0 MeV beam of deuterons from the Ballistic Research Laboratory (BRL) 2.5 MeV Van de Graaff accelerator was used to obtain the RBS and NRS spectra. A Silicon surface barrier detector placed at 160° to the beam direction was used to measure the energies of all scattered charged particles. Since the shapes of the RBS spectra are of critical importance in this work, it was necessary to eliminate the distortions to the spectra which are possible when two pulses from the detector arrive at the analyzer so close in time as to

⁹Anderson, H. H. and Ziegler, J. F., Hydrogen Stopping Powers and Ranges in All Elements, Pergamon Press, New York, 1977.

¹⁰Ziegler, J. F., Biersack, J. P., and Littmark, U., The Stopping and Range of Ions in Solids, Vol. I, Pergamon Press, New York, 1985.

¹¹Semrad, D., Mertens, P., and Bauer, P., "Reference Proton Stopping Cross Section for Five Elements Around the Maximum," Nucl. Inst. Meth., Vol. B15, pp. 86, 1986.

appear as a single pulse with the wrong amplitude. Besides using fast electronics to cut these pileups, the beam currents were kept at 150 nA or less in order to further reduce their effects. The data were collected by a multi-channel analyzer and stored in a VAX 8600 for later analysis.

Two copper samples, two nickel and one aluminum were used in this study. The first Cu sample was a thick foil of 99.999% purity, taken from its sealed plastic shipping sheath and immediately mounted in the chamber. The second Cu sample was a bias sputter plated layer of OFHC copper on a substrate of aluminum. The first Ni sample was a thick sheet mechanically polished, ultrasonically cleaned and rinsed with alcohol and distilled water. The second Ni sample was a nominally 0.76 micron thick self-supporting foil with no special preparation prior to placement in the scattering chamber. The Al sample was 99.1% pure aluminum, mechanically polished, ultrasonically cleaned, and finally sputter cleaned at 3.0 KV in hydrogen and argon.

3. DATA ANALYSIS

The computer program PROFILE,⁸ developed at BRL, was used to analyze the data in this study. PROFILE uses elemental concentrations, stopping powers, and scattering/reaction cross sections as input information in order to produce the expected RBS or NRS distributions. The calculated results are compared to the experimental spectra and the quality of the fit is determined from the match in the shapes of the two distributions. Since elemental concentrations, stopping powers and cross sections can all contribute to a variation in these shapes, it was of utmost importance to eliminate all but the stopping power as a factor in this study. The elemental concentrations were eliminated, or at least much reduced in importance, by choosing thick samples with a minimum of impurities, concentrated only in thin surface layers. The effect of the cross sections was eliminated by performing the experiment in energy regions where the elastic cross sections are expected to be Rutherford and thus well-known. For the cases of deuteron elastic scattering on C and O at energies between 0.5 and 1.0 MeV, the actual rather than Rutherford cross sections were used. Since the actual C and O concentrations were minimal, this use of poorly defined cross sections did not seriously affect the results. The effects of core electron screening on the Rutherford cross sections is ignored since it amounts to less than 1% at the energies involved in this work. Energy loss straggling was included in the calculations but its effects were found to be negligible in the thick target cases. The calculated spectra were broadened by the beam and detector energy resolution functions. In all of the analyses, the amounts of carbon and oxygen were determined from the reaction parts of the data where the (d,p) and (d,alpha) reaction peaks from C and O are found.

An additional factor which may contribute to the yield in RBS distributions is double scattering of the beam particles. Weber and Mommsen¹² have discussed its importance in the case of 400 KeV protons scattered from

¹²Weber, A. and Mommsen, H., "Background in Rutherford Backscattering Spectra-A Simple Formula," Nucl. Inst. Meth., Vol. 204, pp. 599, 1983.

thick gold foils and have shown that as much as 10% of the yield in the lower energy regions of the RBS distributions can be the result of such double scattering. Because this double scattering phenomenon does not lend itself readily to exact calculation and since for 1 MeV deuterons on Al, Ni and Cu the yield is expected to be somewhat lower, it is not included in the present analysis. For similar reasons, multiple scattering has not been included. However, because these effects are not included, the results in terms of absolute fits may not be correct whereas the demonstration of the stopping power effect on the results is still valid.

Figure 1 shows the stopping powers for Al, Ni and Cu from five different sources which were considered in this work. AZ77⁹ is a complete set of proton stopping powers on all elements obtained by the evaluation of all available stopping power data. The results are put into an analytic form with five independent variables which makes it particularly easy to use in an analysis program like PROFILE. This set of stopping powers has been widely used by the IBA community as well as nuclear and atomic physics researchers. ZBL85¹⁰ is a new set which is more complete than AZ77 and is based on a fundamental theoretical model of stopping powers. The results compare very well with the weight of available experimental data, but deviations of more than 10% are common for protons in the energy range of less than 500 KeV. SMB85¹¹ is from a recent measurement of proton stopping powers on Al, Ni, Cu, Ag and Au in the 20 to 700 KeV energy range. Two different techniques were used in two different laboratories using the same samples, with the results combined. These results are also parametrized in the same way as is done in AZ77, making a comparison between the sets within PROFILE quite simple. NS70¹³ was the best available stopping power set until AZ77 was published. SMB85* results from the current work and is the SMB85 set modified so as to yield the best fits to the purest thick targets without compromising the shape of the stopping power curves. As can be seen, the SMB85* deviates from the SMB85 by as much as 5% in the energy range of 100 to 500 KeV. The three sets labeled by SMB85, AZ77 and SMB85* were used in calculating fits to the data. Although Fig. 1 shows proton stopping powers (S_p), the conversion to the deuteron stopping powers (S_d) required in this work is given by $S_d(E) = S_p(\frac{1}{2} E)$.

4. RESULTS AND DISCUSSION

4.1 Copper. Figure 2 (upper) shows the experimental and calculated RBS results for the thick, pure copper sample. Figure 2 (lower) shows the set of elemental concentrations that were used in all three of the calculations. The carbon and oxygen concentrations were determined from the NRS part of the data and are reported in Table 1. The amounts of both C and O are in the 10 to 15 monolayer range. According to the analysis, both were concentrated only in the outer 150 angstroms. These amounts and their distributions are consistent with naturally occurring surface oxidation and some amount of

¹³Northcliffe, L. C. and Schilling, R. F., "Range and Stopping-Power Tables for Heavy for Heavy Ions," Nuclear Data Tables Vol. A7, pp. 233, 1970.

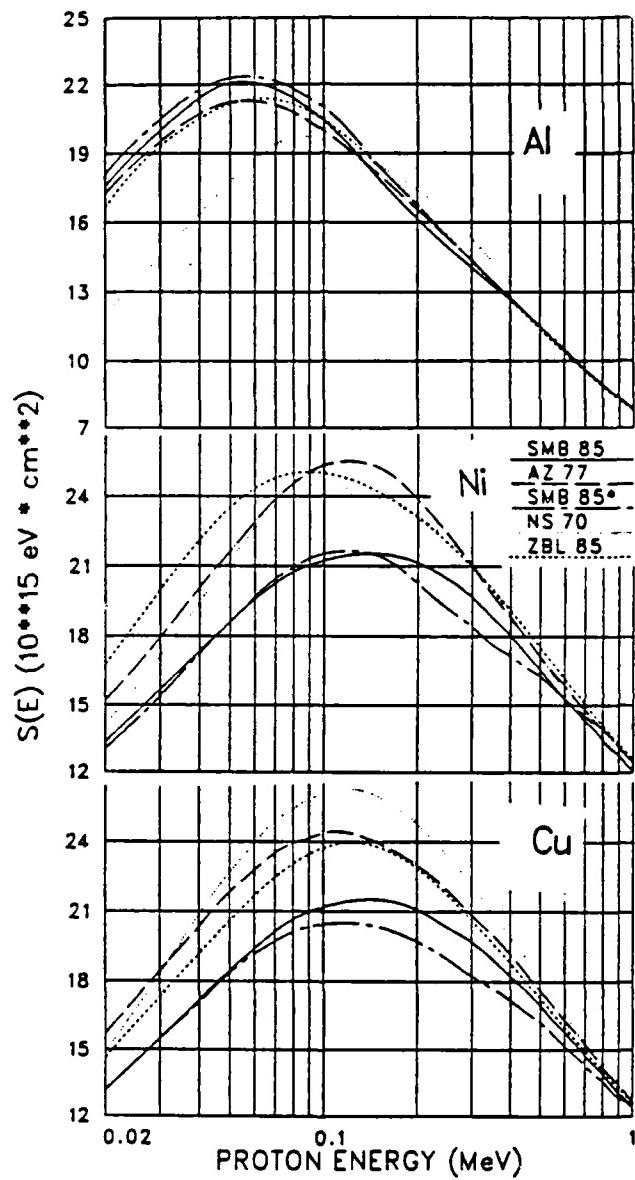


Figure 1. Elemental Stopping Powers for Al, Ni and Cu. Three of the sets, AZ77, ZBL85 and NS70 have been in common use, the SMB85 is from a recent measurement and the SMB85* is from the current analysis.

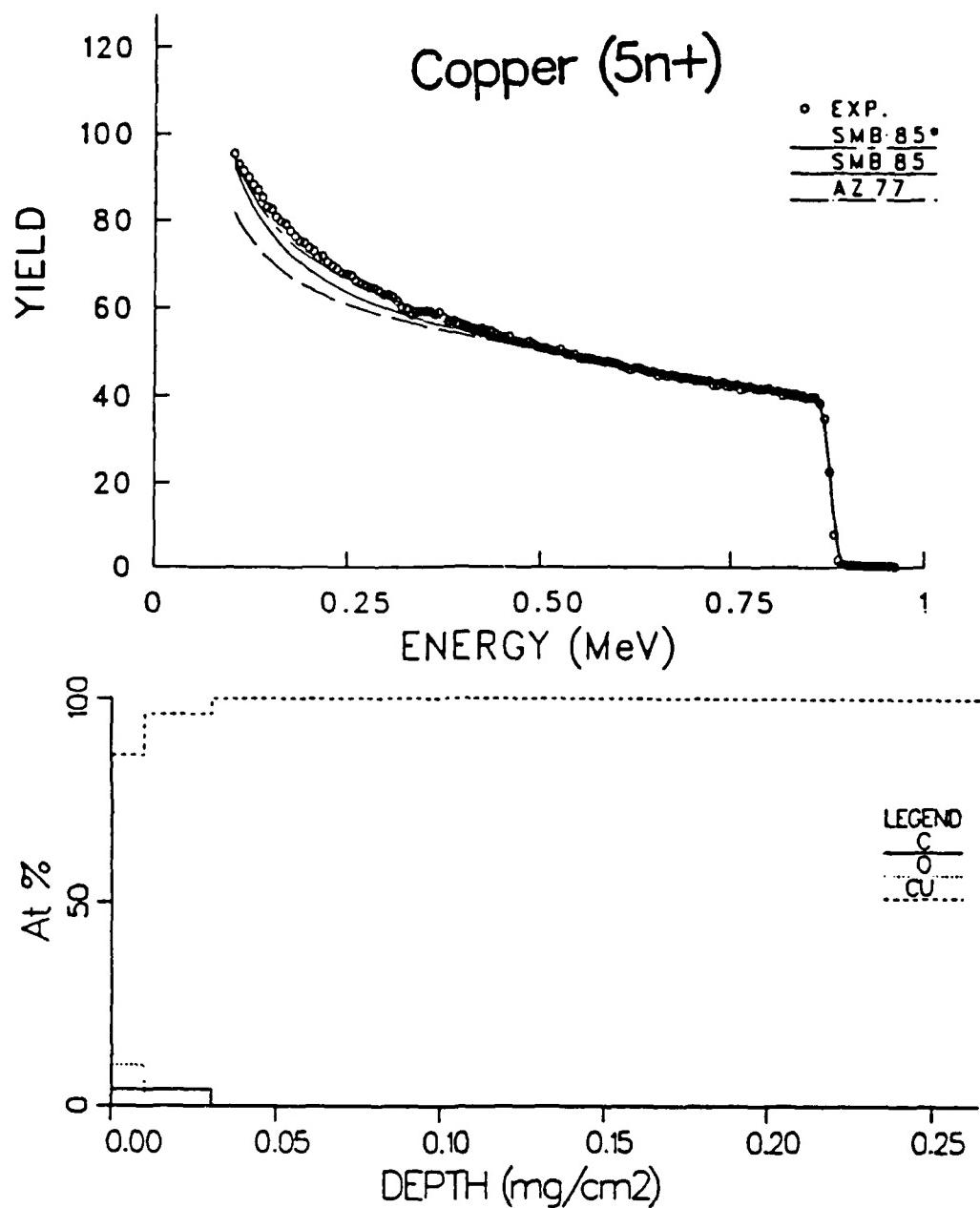


Figure 2. Results for the thick pure copper sample. Along with the data, the upper part shows calculated RBS results with the three stopping power sets as indicated. The lower part shows the elemental concentrations that were used in all three calculations.

carbon buildup from the vacuum system during beam heating of the target spot. In agreement with the supplier's statement concerning its purity, only copper was found in the remainder of the sample.

TABLE 1. Carbon and Oxygen Content on Samples

<u>Sample</u>	<u>Carbon (at/cm²)</u>	<u>Oxygen (at/cm²)</u>
Copper	3.0×10^{16}	2.1×10^{16}
Copper Plate	4.5	2.0
Nickel	4.8	3.5
Nickel Foil	1.9	2.1
Aluminum	3.5	15.0

The RBS data and calculation are normalized by chi-square minimization in the energy range 0.43 to 0.75 MeV. The chi-squares were 1.14 for SMB85*, 4.42 for SMB85 and 8.23 for AZ77. The most significant difference between the three sets is the slope of the RBS distribution. This slope effect can be understood by comparing the differences in the S(E)s from 100 to 500 KeV. All else remaining the same, a higher stopping power will lead to a flatter RBS distribution. It is possible to force the AZ77 to fit the slope more exactly by introducing significant amounts (more than 5%) of an undetected element (such as hydrogen), in a non-constant concentration, deep into the copper sample. However, this would be a clearly unphysical solution. The data and the calculations in this, and all subsequent results, extend down to 100 KeV deuteron energy with the consequence that hydrogen stopping power values to only 50 KeV are used.

4.2 Copper Plate. Figure 3 (upper) shows the RBS results for the copper coating on an aluminum substrate. The three stopping power sets indicated along with the elemental concentrations shown in Fig. 3 (lower) were used in obtaining these fits. As can be seen, the surface carbon and oxygen concentrations (from Table 1) and their depth distributions are similar for this sample and the pure copper sample discussed above. The carbon concentration being about 50% higher is most probably due to the fact that this sample may have been exposed to vacuum system hydrocarbons for a longer period of time. The additional carbon at the interface between the copper and aluminum may be due to the incomplete cleaning of the starting aluminum surface prior to the beginning of the ion plating.

The RBS analysis on this sample illustrates the capability of this technique for determining the coating thickness. The Cu layer thickness, determined by measuring the mass of the coating, averaged over the sample area, was 1.20 mg/cm^2 , the AZ77 fit required 1.17 mg/cm^2 and the SMB85* fit required 1.37 mg/cm^2 . Since the corners and edges of the sample had visibly

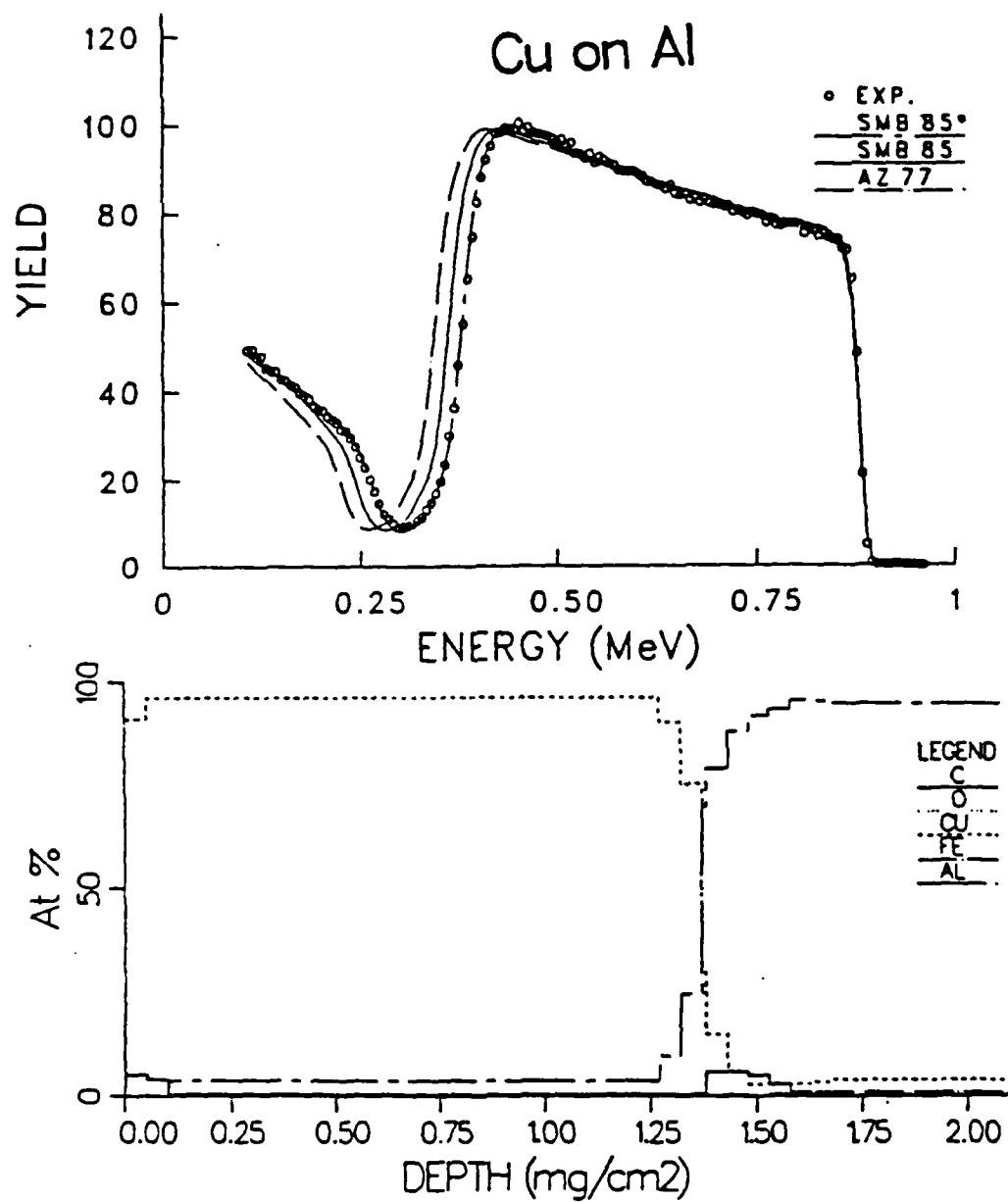


Figure 3. Results for the Cu plated Al sample. See Fig. 2 caption for the description of the two parts. A small amount of carbon can be seen in the aluminum surface at the interface between the copper and aluminum.

less copper coating than the center where the beam hit the sample, the 1.20 mg/cm^2 is clearly an underestimate of the central thickness. In addition, the SMB85* gives the best fit to the slope of the distribution, yielding a chi-square of 1.75 whereas SMB85 gives 4.72 and AZ77 gives 8.35. The fact that some aluminum was found throughout the copper layer is explained by the plating configuration. The sample was being held in an aluminum holder during the ion plating. Since this plating process sputtered the sample and its holder during the deposition phase, some of the aluminum from the holder was deposited along with the copper.

The AZ77 stopping power values could be used to obtain as good a fit as the SMB85* gives only if several important changes are allowed. First, the thickness of the copper layer must be decreased by about 8% in order to make the width of the copper distribution match the experimental data. As was seen from the above discussion, this decrease in the thickness of the central part of the sample is not consistent with the mass measurement and non-uniformity of the coating. Second, a small amount of hydrogen, at the 1% level, must be distributed in the copper layer so that the calculated slope matches the experimental data. Requiring hydrogen at the 1% level throughout the greater than 1mg/cm^2 depth of the copper layer is also unlikely. A number of other samples, plated under similar conditions, were analyzed for hydrogen at SUNY Albany by W.A. Lanford, using a ^{15}N beam and the method he described in Ref.¹⁴ In all cases the concentration of H did not exceed 1% of the surface and decreased rapidly to less than .1% in a depth of $.02\text{mg/cm}^2$.

4.3 Nickel. Figure 4 (upper) shows the RBS results for the thick nickel slab. The three stopping power sets indicated along with the elemental concentrations shown in Fig. 4 (lower) were used. It can be seen that the difference between the SMB85* and SMB85 stopping power sets are similar in the Ni and Cu cases, namely, that they deviate by about 5% in the 150 to 400 KeV energy range. This produces a similar result in the RBS analysis, namely, that the major difference between the different stopping power sets is the slope of the calculated RBS distributions. The normalization is again over the energy range of 0.43 to 0.75 MeV yielding chi-squares of 3.58 for SMB85*, 15.40 for SMB85 and 17.83 for AZ77. The reason for the much poorer fits to the nickel than the copper data may be explained by the relatively larger amount of oxygen on the nickel surface.

4.4 Nickel Foil. Figure 5 (upper) shows the RBS and Fig. 5 (middle) shows the NRS results for a nominally 0.78 micron or 0.678 mg/cm^2 thick Ni foil. The elemental profiles shown in Fig. 5 (lower) are used in both cases. The average thickness of this foil was determined to be $0.642(+/- 0.01)\text{mg/cm}^2$ by weight and area measurements. The RBS analysis gave a thickness of 0.64mg/cm^2 at the location of the beam spot. A small amount of oxygen existed on both

¹⁴Lanford, W. A., Trautvetter, H. P., Ziegler, J. F., and Keller, J., "New Precision Technique for Measuring the Concentration Versus Depth of Hydrogen in Solids," Appl. Phys. Lett. Vol. 28, pp 566, 1976.

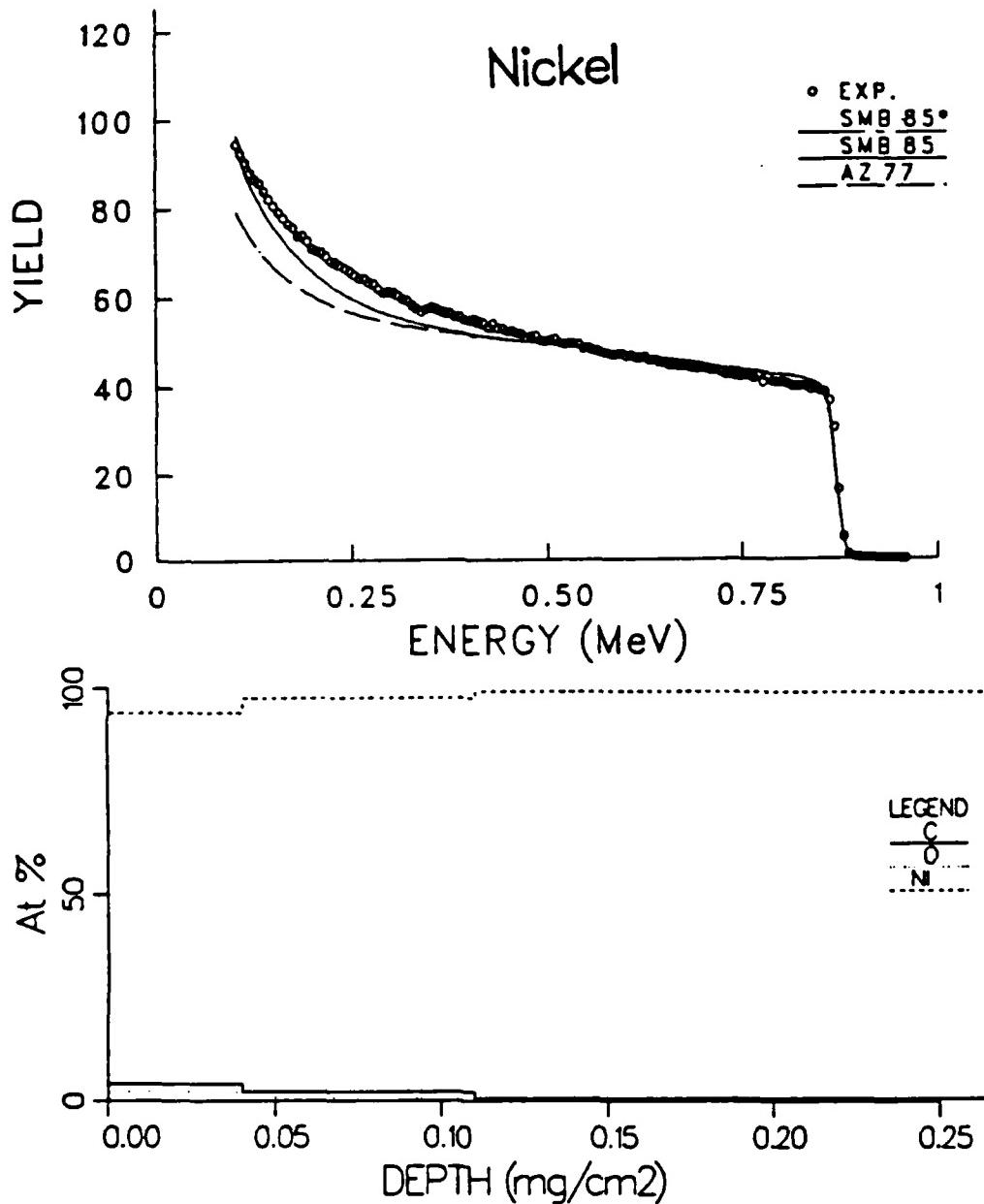


Figure 4. Results for the thick Ni sample. See Fig. 2
caption for the description of the two parts.

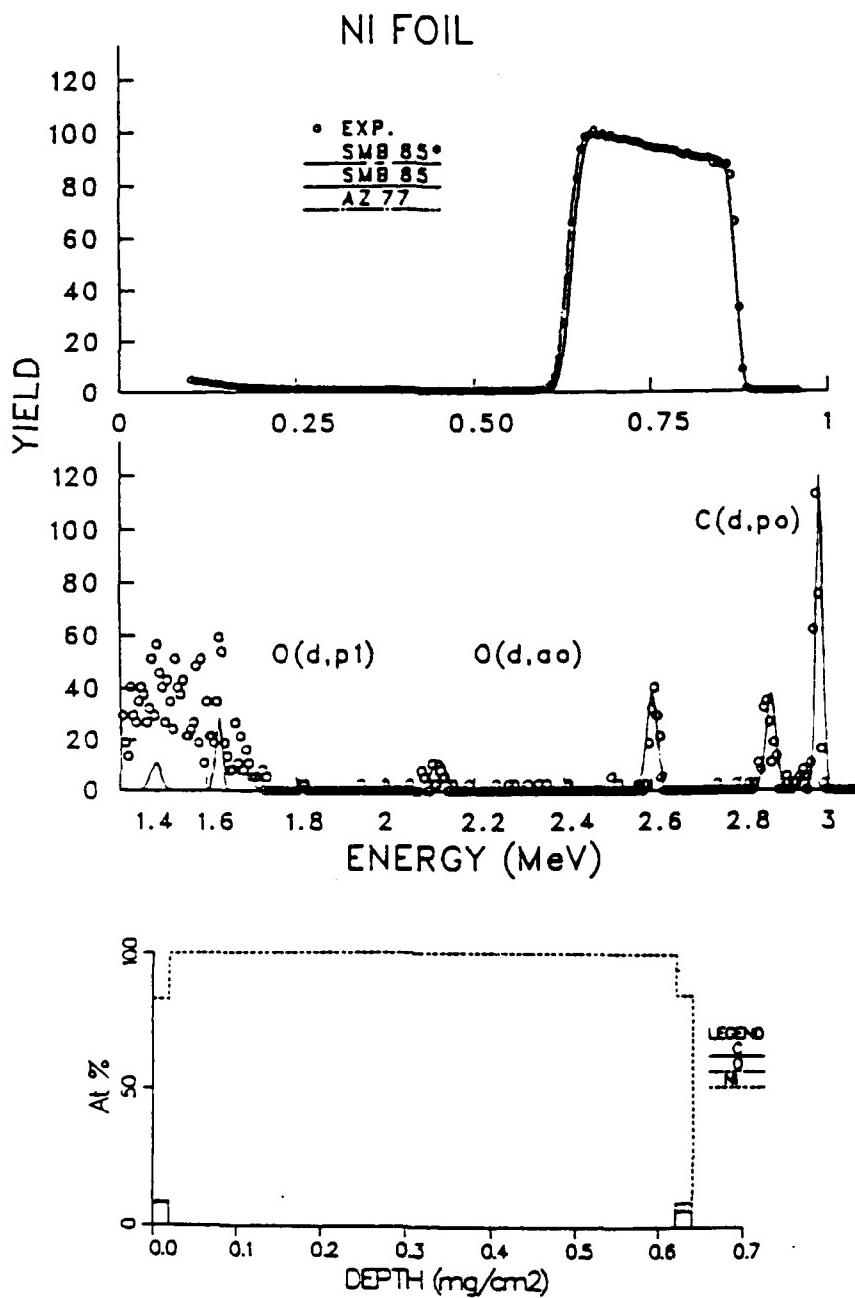


Figure 5. Results for the Ni foil sample. In this case, the NRS results are shown in the middle along with the RBS at the top and the elemental profiles at the bottom. The (d,α) peaks can be used as a very accurate gauge of the foil thickness.

surfaces of the foil as can be seen from Fig. 5 (middle). This fact allowed the (d, α) reaction on O to be used for determining the thickness of the foil more precisely than could be done by elastic deuteron scattering from Ni. As the Ni foil thickness used by the PROFILE analysis program is increased, the distance between the two (d, α) peaks as calculated also increases. A change of 0.01 mg/cm^2 is enough to cause a noticeable degradation of the fit, so the uncertainty on the foil thickness is this same 0.01 mg/cm^2 .

Although it appears that there is not much difference between the RBS fits with the three stopping power sets, the chi-squares do show a significant change. They were 1.23 for SMB85*, 14.73 for SMB85 and 4.13 for AZ77. The reason for the very poor value with SMB85 is that the Ni stopping powers are not well defined by the original data in the energy region above 500 KeV, where they are most needed in analysis of this particular sample.

4.5 Aluminum. Figure 6 (upper) shows the RBS results for the thick Al sample using the elemental profiles shown in Fig. 6 (lower). The aluminum normalization is done from 0.25 to 0.70 MeV and the chi-squares are 1.26 for the SMB85*, 2.27 for SMB85 and 1.26 for AZ77. All of the difference in the three calculations occurs below 0.25 MeV, a result which is consistent with the differences in the S(E)'s shown in Fig. 1. From Table 1 it can be seen that this is the only sample in this experiment which contains more than just a nominal amount of oxygen. This is consistent with the fact that an aluminum surface oxidizes more readily than copper or nickel.

The foregoing results on the five samples is indicative of the sensitivity of the RBS method of surface analysis to uncertainties in the elemental stopping powers. It was found that even a 1% variation of a stopping power in a limited energy range was sufficient to cause a significant and observable change in the RBS results. The consequence of this is that surface constituent and coating thickness determinations by RBS can be in error unless absolutely correct values of stopping powers are used.

It is seen that combining detailed computer analysis of RBS spectra on pure elemental samples may be capable of determining the stopping power curve over a fairly large energy range, say from 50 KeV to 1.0 MeV, in a single experiment. However, the SMB85* stopping powers for Al, Ni and Cu are not proposed as the correct values. In order to use this type of RBS analysis to in fact determine stopping powers, much more careful experiments and calculations need to be performed. First, in-situ sample cleaning procedures should be available so that even the small amounts of oxygen and carbon could be eliminated from the surfaces. Secondly, the samples that are used should be certified pure at the 99.9% level at least. Although this condition was met by the thick copper sample used in this experiment, such was not the case with any of the others. Third, care must be exercised in orienting any of the samples that are used to avoid orientations which can give rise to channeling effects. Fourth, since pulse pile-up and detector noise can be very significant factors in distorting the shapes of the RBS distributions, they must be totally eliminated. This was only partially accomplished in the present work. Finally, all possible sources of RBS yield must be included in the calculation, a requirement that up to now has not been feasible.

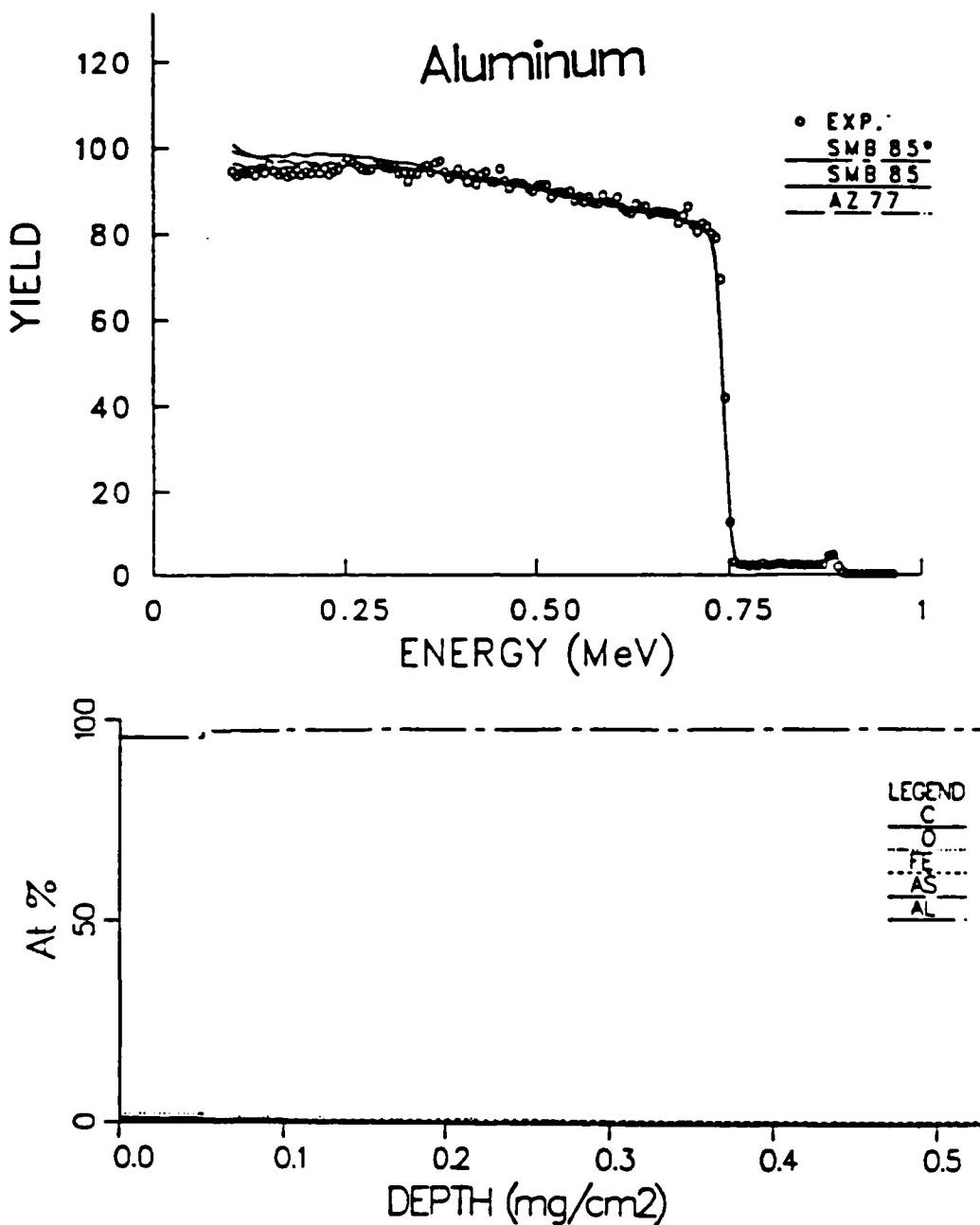


Figure 6. Results for the thick Al sample. See Fig. 2 caption for the description of the two parts.

5. CONCLUSIONS.

The analysis of thick target RBS distributions from Cu, Ni and Al clearly shows that accurate stopping powers are important to the accurate determination of the concentrations of elements on the target surface. The 15% uncertainties in the region of the stopping power maximum have an effect of similar magnitude in the determination of coating thicknesses and can result in serious errors in the identification of elements present in the target. When proper care is exercised in eliminating uncertainties from thick target RBS data, and the calculation used to simulate the experiential spectrum is indeed complete, stopping powers can be determined simply and accurately.

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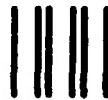
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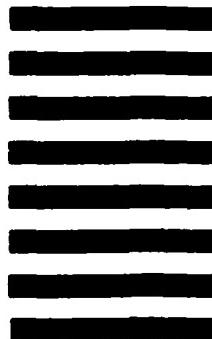


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